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Effect of Cleanliness on Hydrogen Tolerance in High-Strength Steel

by Scott M. Grendahl, Franklyn Kellogg, and Hoang Nguyen

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Effect of Cleanliness on Hydrogen Tolerance in High-Strength Steel

Scott M. Grendahl
Weapons and Materials Research Directorate, ARL

Franklyn Kellogg and Hoang Nguyen
Bowhead Technical Services

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14. ABSTRACT
This work utilized a Design of Experiments (DoEs) approach to generate comparative data sets for the common test geometries across a range of load level, material strength, and hydrogen concentration. The design created an empirical predictive mathematical model for failure based on input conditions. The models have proven successful in qualitative and quantitative assessment of performance. Targeted models will be developed for prospective maintenance chemicals and coatings where implementation is currently hindered because of hydrogen concerns. These models will clearly show where the materials can be safely employed based on material strength and/or loading level in the application. Alleviation of hydrogen concerns for popular chemicals and coatings will lead to implementation at military depots and the commercial aerospace industry using new, more environmentally friendly solvents and coatings.

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1. Introduction

Solvent substitution for maintenance and overhaul operations of military systems has been a primary environmental concern for many years. Cadmium replacement in these systems has been targeted for decades. Both of these areas have a common obstacle for implementation of any potential alternative. Hydrogen embrittlement of high-strength steel is the most predominant unforeseen hurdle because high-strength materials show sensitivity to the phenomena and the source of the hydrogen can be anything within the fabrication process, maintenance practice, or the natural corrosion cycle. Standardized testing on this issue has traditionally stemmed from the aerospace industry where it is a principal focus. Historically, the various aerospace defense contractors have each tested in their own manner, which has led to the national standard incorporating many approved test geometries and "grey" procedures. This standardized test is a "pass/fail," "go/no-go" type of test and the "grey" procedures lead directly to conflicting test results and perceived risks and/or roadblocks when it comes to implementation of proposed alternative chemicals and coatings. This work evaluated hydrogen susceptibility over a range of material strength, load level, and hydrogen emitting environment (weight-percent [wt.%] sodium chloride [NaCl]) in two different materials grades of 4340, while developing life-predictive models for each geometry. This demonstrated the comparative susceptibility of each material and should greatly increase the applications for which they will be considered, as the models provide the acceptability criteria for the parameters specific to each application.

2. Objective

This work was designed to utilize a Design of Experiment (DoE) approach to create life-prediction models for air-melted SAE-AMS-6415 and vacuum-melted SAE-AMS-6414 (aerospace grade) steel using common American Society for Testing and Materials (ASTM) F-519 specimen geometries in combination with load-cell measurement and time-monitored experiments.[1–3] The data gathered will allow a conclusive determination to be made as to which material is more sensitive to hydrogen. The method will be subsequently used to evaluate the most prospective environmentally friendly maintenance chemicals and cadmium-alternative coatings that currently have their use limited via the perceived risk of hydrogen embrittlement.

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[1] SAE-AMS-6415S-2007. Steel, Bars, Forgings, and Tubing 0.80Cr - 1.8Ni - 0.25Mo (0.38–0.43C), SAE World Headquarters, Warrendale, PA, 2007.

[2] SAE-AMS-6414K-2007. Steel, Bars, Forgings, and Tubing 0.80Cr - 1.8Ni - 0.25Mo (0.38–0.43C) (SAE 4340) Vacuum Consumable Electrode Remelted, SAE World Headquarters, Warrendale, PA, 2007.

[3] ASTM F 519-10. Standard Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating/Coating Processes and Service Environments, ASTM International, West Conshohocken, PA, 2010.

3. Materials

The five specimen geometries used, fabricated from both 6415 air-melted and 6414 aerospace 4340 steel, were manufactured in accordance with the geometries of ASTM-F-519 1a.1, 1a.2, 1c, 1d, and 1e specimens. These specimens are commonly used by nearly all of the aerospace industry and technical community for conducting hydrogen embrittlement research. They are depicted in figure 1.

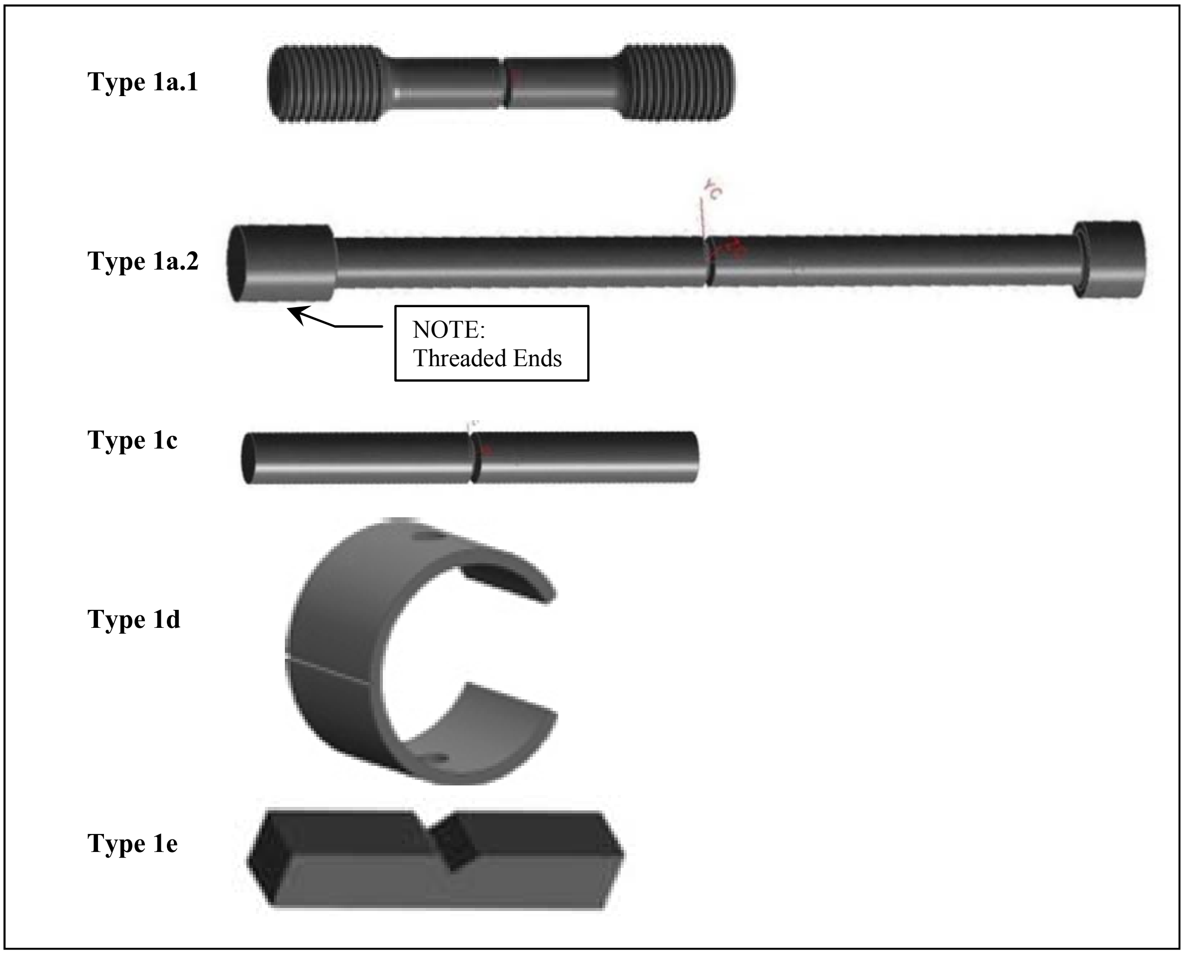

Figure 1. ASTM-F-519 specimen geometries.

3.1 Heat Treating

A critical element in conducting this comparative research across the five geometries was to have the material strength as close to identical as possible. This proved tedious as the stock removal differs on each specimen geometry in blanking and final machining. Additionally, production heat treating proved an imprecise process without tight control. Suppliers were not used to keeping such tight tolerances on their heat-treated product. It was crucial to have the strength level of each specimen in a very narrow range (± 5 ksi); otherwise data variation based on geometry might not be observable in the output. The team constructed a submatrix for the background work. This process entailed certification of a rack-basket, hardening furnace, and tempering furnace by normalizing, hardening, and tempering samples to 280 ksi by utilizing small cylindrical buttons for inprocess hardness tests and verification tensile samples. Once tested, verified, and certified per mutually agreed parameters, furnaces and ovens had the process frozen for approval. The heat treatments of the actual specimens were completed within 30 days of the date of frozen planning approval. There were five heat treatment batches for this work across five (5) ASTM-F-519 specimen geometries—1a.1, 1a.2, 1c, 1d, and 1e. Each batch of specimens, T1–T5, required heat treatment in accordance with the following:

- T1 = 140 ± 5 ksi (135–145 ksi)
- T2 = 158 ± 5 ksi (153–163 ksi)
- T3 = 210 ± 5 ksi (205–215 ksi)
- T4 = 262 ± 5 ksi (257–267 ksi)
- T5 = 280 ± 5 ksi (275–285 ksi)

The specimen counts varied by temper level following the overall DoEs. The specimens were heat treated in batches according to their temper lot designation depicted in table 1. The individual quantities were derived from the DoE matrix further explained in the experimental procedures section.

- T1 = 30 + 6 tensiles
- T2 = 75 + 6 tensiles
- T3 = 180 + 6 tensiles
- T4 = 75 + 6 tensiles
- T5 = 45 + 6 tensiles

Table 1. Temper lot quantities.

Temper Lot No.	Strength Target (ksi)	No. of Specimens							
		1a.1	1a.2	1c	1d	1e	Total	+	Tensiles
T1	140	6	6	6	6	6	30	+	6
T2	158	15	15	15	15	15	75	+	6
T3	210	36	36	36	36	36	180	+	6
T4	262	15	15	15	15	15	75	+	6
T5	280	9	9	9	9	9	45	+	6

3.2 Cadmium Plating

The plating requirements were critical because the surface area plated affects both the amount of hydrogen introduced into the sample and the free path out of the sample during hydrogen embrittlement (HE) bake relief. Specimens were supplied in the stress relieved condition to an aerospace industry-approved cadmium-plating vendor. The cadmium plating was low hydrogen embrittlement (LHE) cadmium in accordance with MIL-STD-870 Rev. C. Type II, Class 1. The threads were masked and the specimens were postprocessed baked at 375 ± 25 °F within 1 hour (h) of plating. Plating requirements were set so that each specimen would have an equivalent surface-area-to-volume ratio during environmental testing, but were largely dependent on the allowable container size for holding the test fluid. The plating requirements were set such that no fluid would contact bare unplated steel during testing. The plated area of the specimens was in accordance with figure 2.

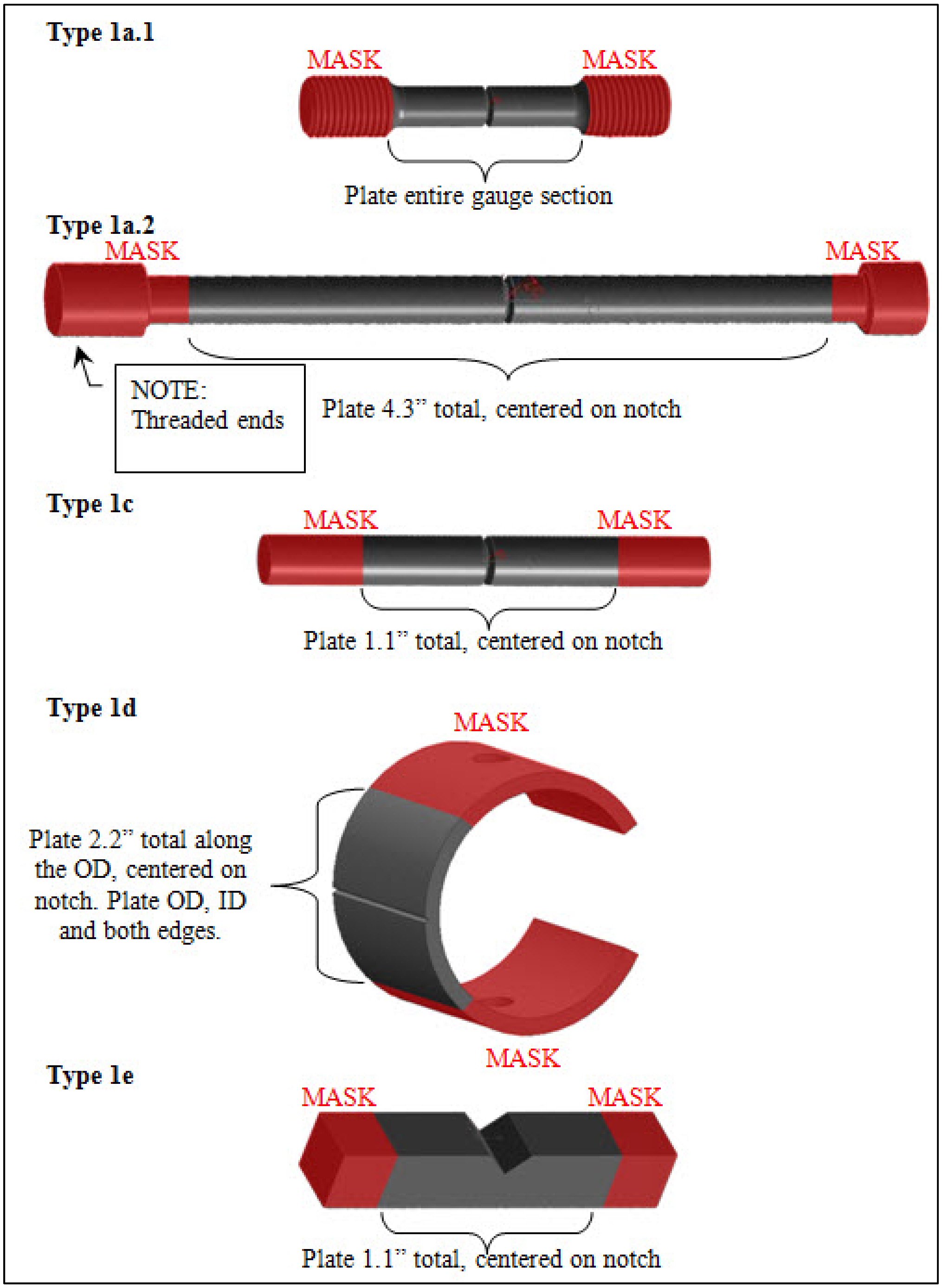

Figure 2. Masking/plating of the five specimen geometries.

4. Experimental Procedures

4.1 Design of Experiment

This approach was used over a range of material strength for both grades of 4340 steel, load level, and hydrogen environment. The five geometries were tested while load levels were monitored to determine a precise time to fracture at specific percentages of notch fracture strength (NFS), specific material strengths (heat-treat tempers T1–T5), and specific hydrogen-emitting environment (sodium chloride, wt.% NaCl). Conversely to the existing standard, greater information was gleaned beyond the result of a pass/fail test. By incorporating the failure time, load, and stress-level data into DoE failure models, predictive equations over the broad ranges were developed.

The DoE focused on three (3) variables for the five (5) geometries (ASTM-F-519 types 1a.1, 1a.2, 1c, 1d, and 1e). The control variables were selected from risk reduction and ruggedness leveraged efforts conducted by the Boeing Company with the assistance of the U.S. Army Research Laboratory (ARL). The five geometries were selected from the ASTM-F-519 test method. Table 2 presents the range of test conditions for the five ASTM-F-519 test geometries researched.

Table 2. DoE conditions matrix.

Condition	–α	—	0	+	+α
Strength (ksi)	140	158	210	262	280
Test Load (% NFS)	40	45	60	75	95
NaCl Concentration (wt.% NaCl)	1.25E-05	0.01	0.50	2.36	3.5

Below 140-ksi steel is generally accepted as not being sensitive to hydrogen, which set the lower limit for strength. NaCl was not used at 0%, essentially completely deionized water, since the working group had experience that deionized water is actually severely corrosive and a very harsh environment for steel. It is also not a real-world environment.

The DoE approach was refined with preliminary ruggedness and risk reduction efforts at Boeing Mesa, with technical assistance from Boeing St. Louis, Seattle, and ARL. Typical of DoEs, it consisted of three test portions—a linear portion, a quadratic portion, and a confirmation portion. The example matrix is as presented in tables 3–5 with the condition values corresponding to table 2. These experiments aided the development of appropriate boundary conditions for the larger effort.

Table 3. Linear portion test matrix.

Repeat Entire Matrix 2x for 1a.1, 1a.2, 1c, 1d, and 1e	—	A	B	C	Run Order
	RUN ID	**Strength (ksi)**	**Test Load (%NFS)**	**NaCl Conc. (wt.% NaCl)**	
Linear Portion	L1	—	—	—	Random
	L2	—	—	+	
	L3	—	+	—	
	L4	—	+	+	
	L5	+	—	—	
	L6	+	—	+	
	L7	+	+	—	
	L8	+	+	+	
Center Points	C1	0	0	0	
	C2	0	0	0	
	C3	0	0	0	
	C4	0	0	0	
	C5	0	0	0	
	C6	0	0	0	

Table 4. Quadratic portion test matrix.

	—	A	B	C	Run Order
Repeat Q1–Q6 5x for 1a.1, 1a.2, 1c, 1d, and 1e	**RUN ID**	**Strength (ksi)**	**Test Load (%NFS)**	**NaCl Conc. (wt.% NaCl)**	
Not Replicated	C7	0	0	0	First
Quadratic Portion	Q1	$+\alpha$	0	0	Random
	Q2	$-\alpha$	0	0	
	Q3	0	$+\alpha$	0	
	Q4	0	$-\alpha$	0	
	Q5	0	0	$+\alpha$	
	Q6	0	0	$-\alpha$	
Not Replicated	C8	0	0	0	Last

Table 5. Confirmation portion test matrix.

—	—	A	B	C	Run Order
	RUN ID	**Strength (ksi)**	**Test Load (%NFS)**	**NaCl Conc. (wt.% NaCl)**	
Confirmation Portion	1	Varied depending on outcome of linear, center, and quadratic			Random
	2				
	3				
	4				
	5				
	6				
	7				
	8				
	9				
	10				
	11				
	12				
	13				
	14				
	15				
	16				

After the linear and center point data runs are completed, initial calculations were made for the predictive model equations. Those initial models were utilized to choose confirmation runs to be researched. The confirmation run results were then incorporated into refining the initial working model.

4.2 Specimens, Environments, and Loading Procedure

Air-melted and aerospace grade 4340 steel samples in five ASTM-F-519 geometries and heat treated to five different material strengths, as previously described, were tested. The specimens demonstrated adequate hydrogen sensitivity of the material conducted in accordance with ASTM-F-519. The cadmium-plated specimens used for these experiments are depicted in figure 3.

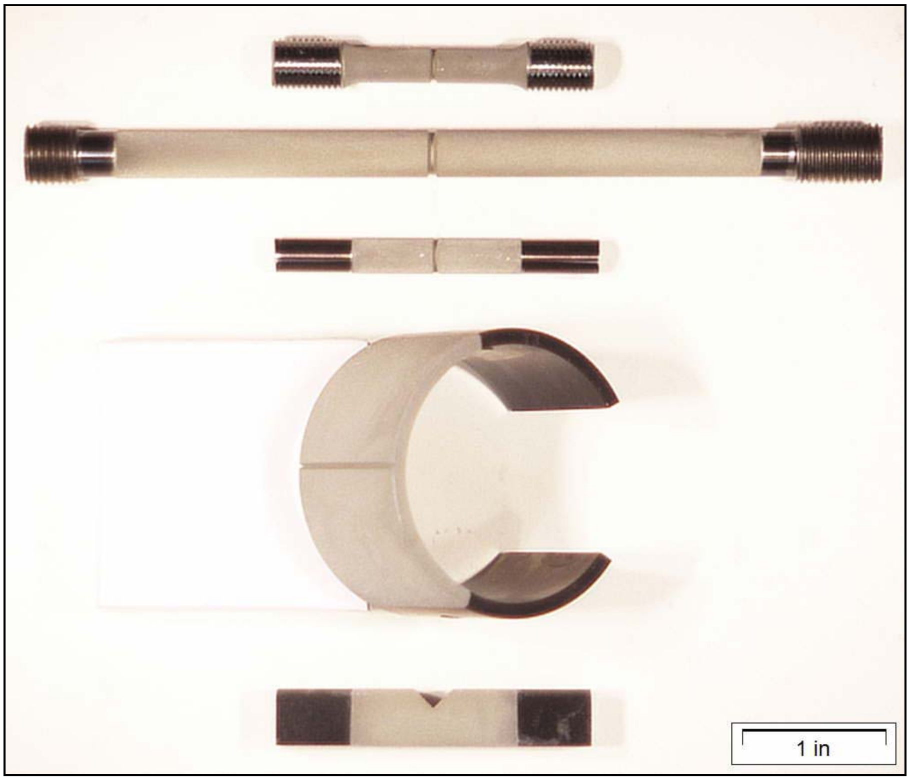

Figure 3. Cadmium-plated experimental specimens (top to bottom: 1a.1, 1a.2, 1c, 1d, and 1e).

The axially loaded specimens (geometries 1a.1 and 1a.2) were tested on Instron* or MTS† uniaxial load-mechanical test frames; the 1c and 1e specimens were loaded with double-cantilever bending fixtures; and the 1d specimens were directly loaded with nut and bolt. The loads were monitored with the load cells on the mechanical test frames and via loading rings installed in the load path for the other geometries. The load cells and load rings were calibrated prior to the experiments. For this work, loads were applied as a percentage (45%–95%) of the calculated 100% NFS determined for each geometry. Ten specimens were utilized to calculate the average 100% NFS with the identical fixturing applied during the experiments. Ten specimens from each group were loaded to failure. The experimental loading was then applied as a percentage of this determined average NFS failure load. Loads were recorded from the mechanical test frames for geometries 1a.1 and 1a.2 and with data-sampling hardware and software for the other geometries. Figures 4–7 depict the insitu test apparatus for the experiments.

The samples were cadmium plated at Asko Processing, Inc., Seattle, WA, in accordance with MIL-STD-870 Rev. C. Type II, Class 1. Plated samples were sensitivity tested in accordance with ASTM-F-519. Cadmium-plating process embrittlement testing involved loading three T5

*Instron is a registered trademark of Illinois Tool Works Inc.

†MTS is a registered trademark of MTS Systems Corporation.

samples from each geometry to 75% of their NFS and holding for 200 h in air. These specimens did not fail, and thus insured that the plating process did not embrittle the specimens.

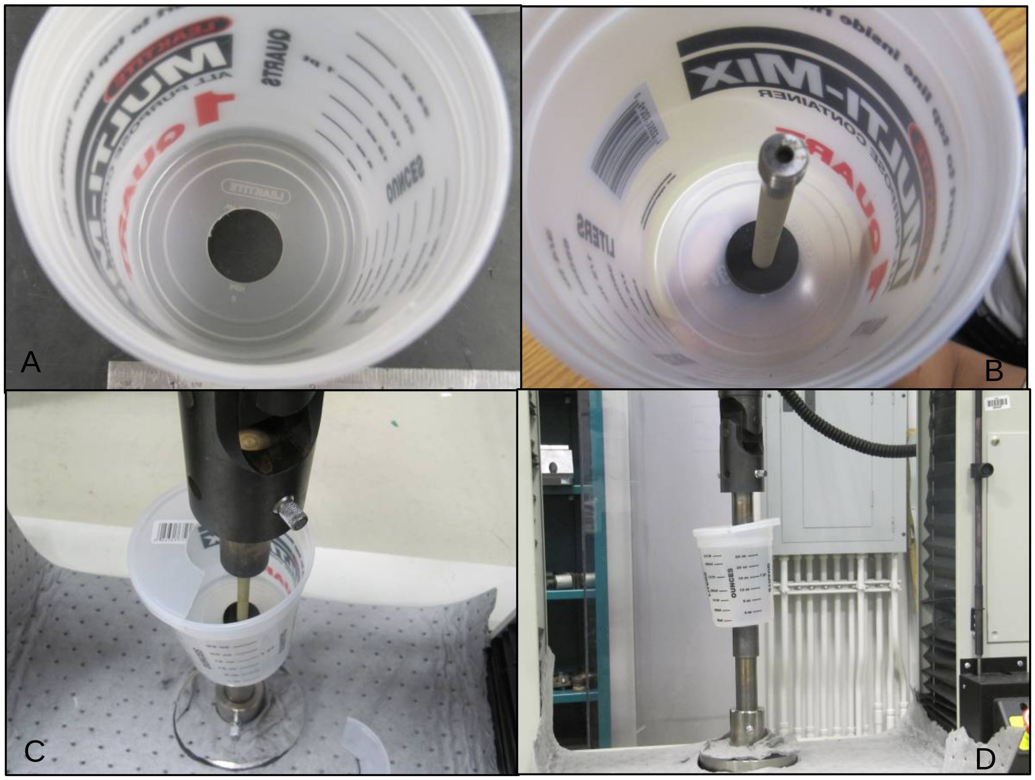

Figure 4. Geometry 1a.1 and 1a.2 insitu environmental setup: (a) the empty container, (b) the sample in the cup, (c) the sample loaded onto the mechanical test frame, and (d) the sample being tested in solution.

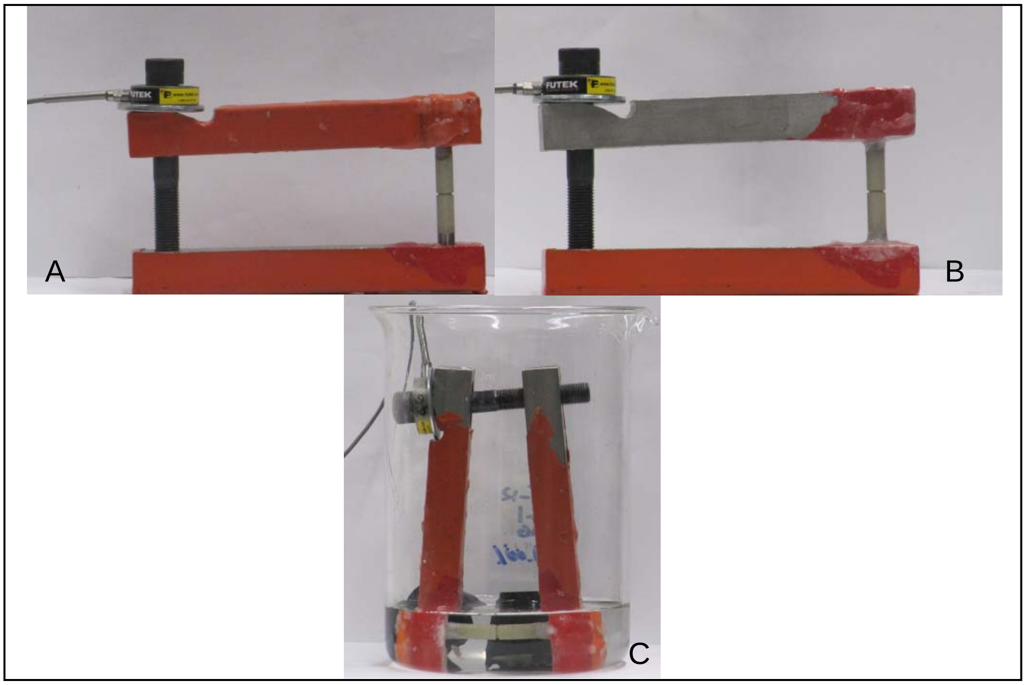

Figure 5. Geometry 1c insitu environmental setup: (a) loaded, (b) loaded and masked, and (c) being tested in solution.

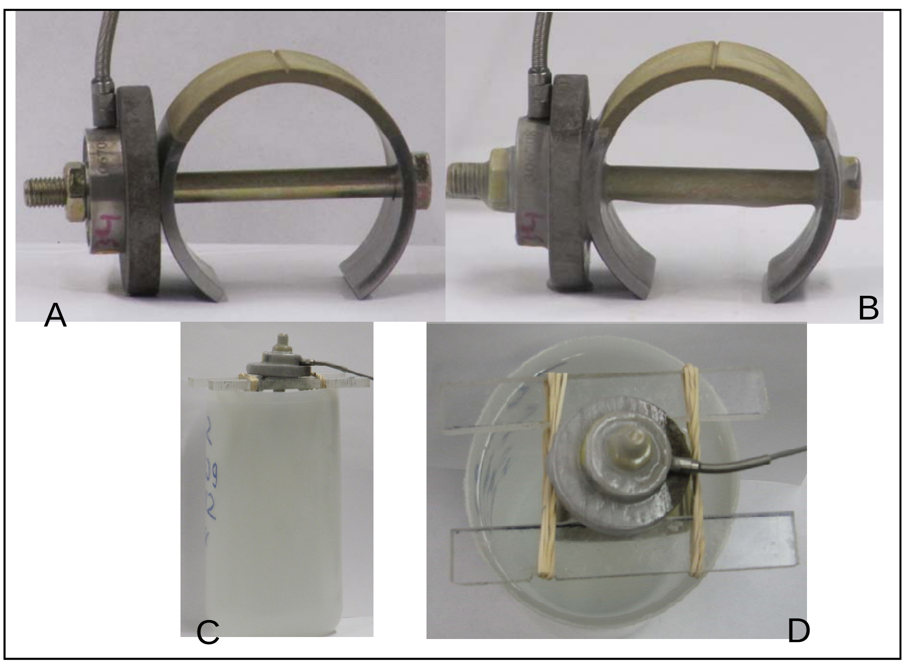

Figure 6. Geometry 1d insitu environmental setup: (a) loaded, (b) loaded and masked, (c) being tested in solution, and (d) top-down perspective.

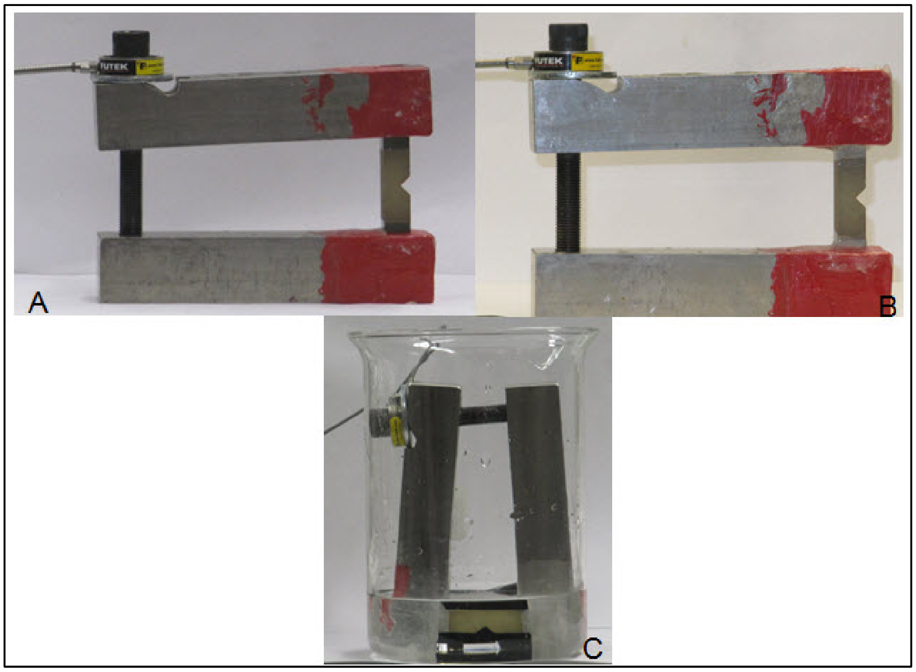

Figure 7. Geometry 1e insitu environmental setup: (a) loaded, (b) loaded and masked, and (c) being tested in salt water.

The specimens were masked so that only the cadmium-plated surface contacted the test solution. The solution used was NaCl in deionized water in five different concentrations: 1.25e-5 wt.%, 0.01 wt.%, 0.5wt.%, 2.36 wt.%, and 3.5 wt.% in accordance with table 2. The volume of NaCl solution for each sample geometry was calculated to ensure that each geometry had the same ratio of cadmium-plated surface area to solution volume (if this volume was not enough to submerge the samples adequately, clean inert material was added to displace solution in order to submerge the samples to the correct level). The loaded specimens were then immersed in the test solution for the duration of the experiment. Specimens were removed either upon failure or after 168 h of sustained load without failure.

As stated previously, upon conclusion of the linear, center, and quadratic test runs preliminary life-prediction models were created. These models were then used in the confirmation test portion of the matrix to choose appropriate parameters to both enhance and verify the model. Final life-prediction equations and three-dimensional models were created after the incorporation of the confirmation data.

5. Results

The final life-prediction models for each geometry and material are presented in figures 8–12 for types 1a.1, 1a.2, 1c, 1d, and 1e respectively. The models, for each respective geometry and material, did not vary significantly from the preliminary set, thus verifying the initial prediction and methods.

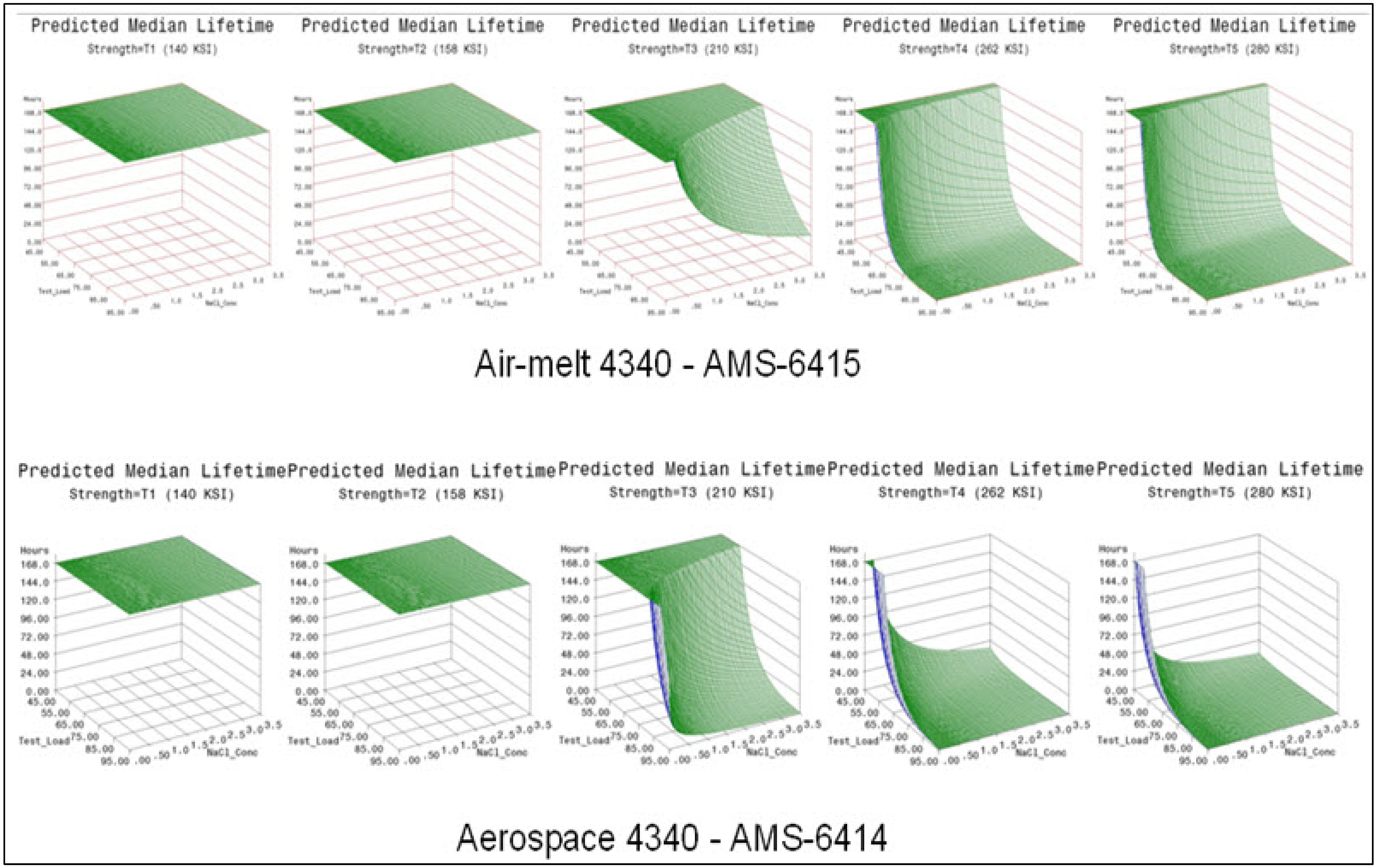

Figure 8. Final 1a.1 specimen geometry life-prediction models.

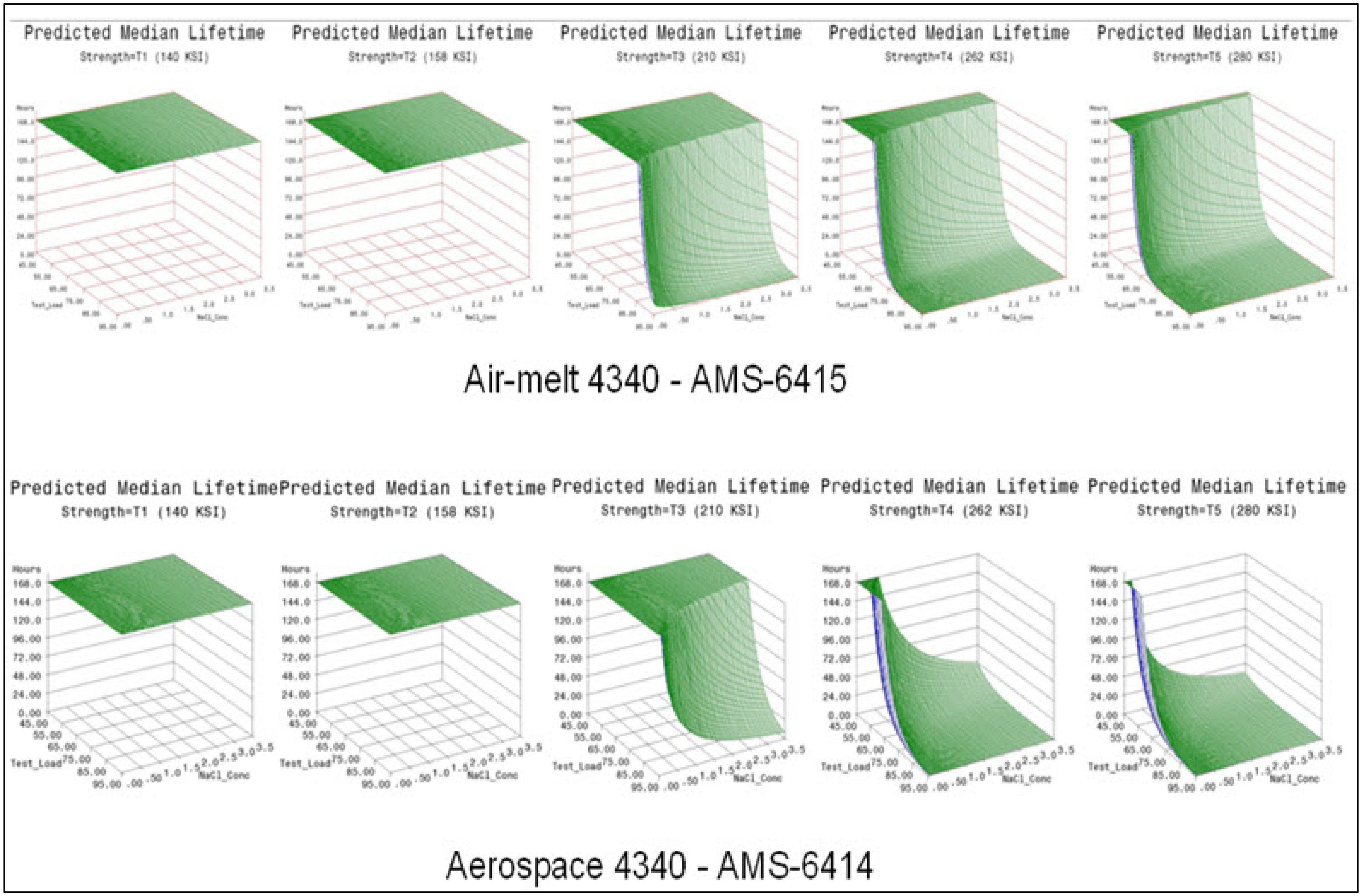

Figure 9. Final 1a.2 specimen geometry life-prediction models.

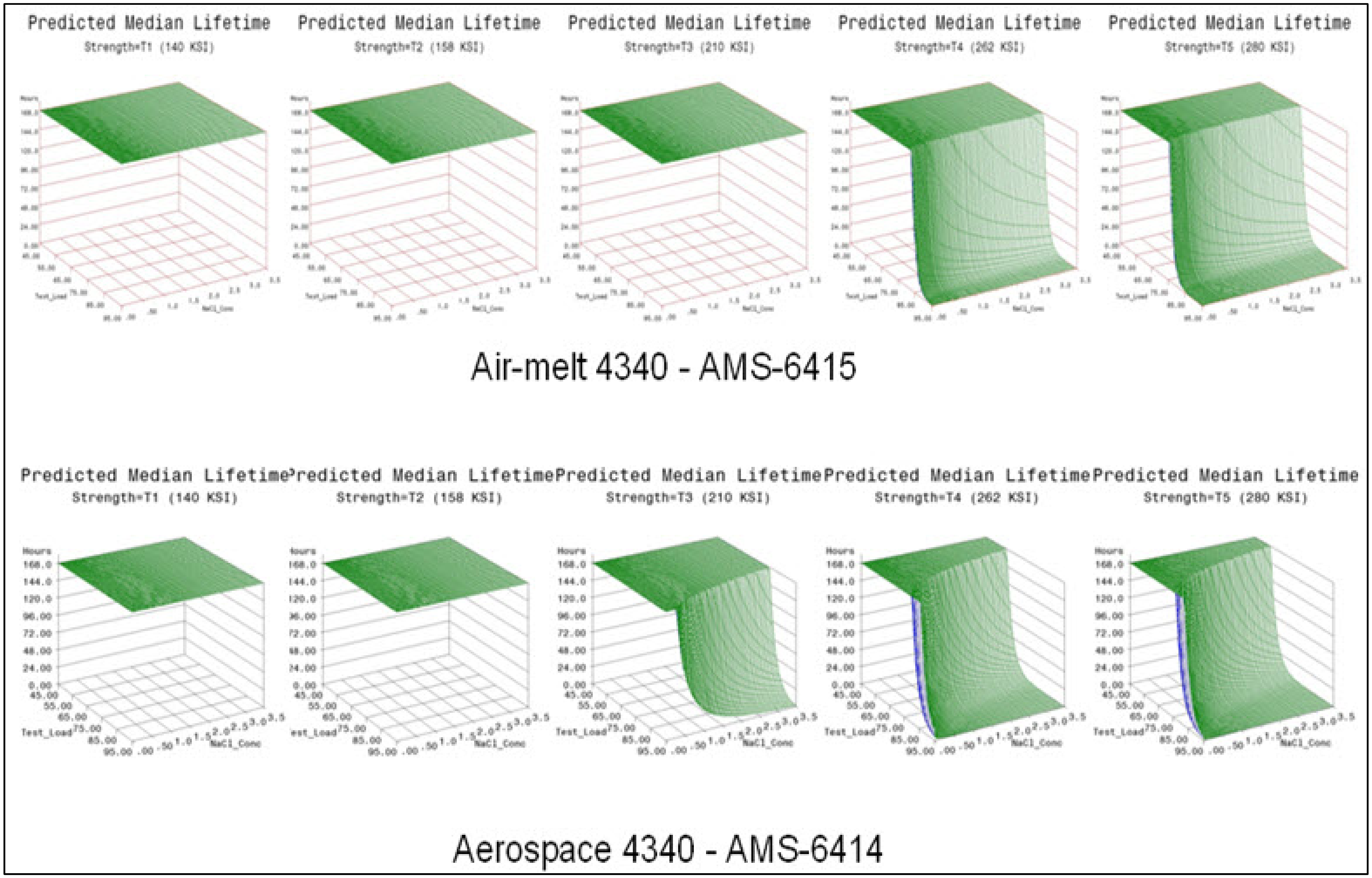

Figure 10. Final 1c specimen geometry life-prediction models.

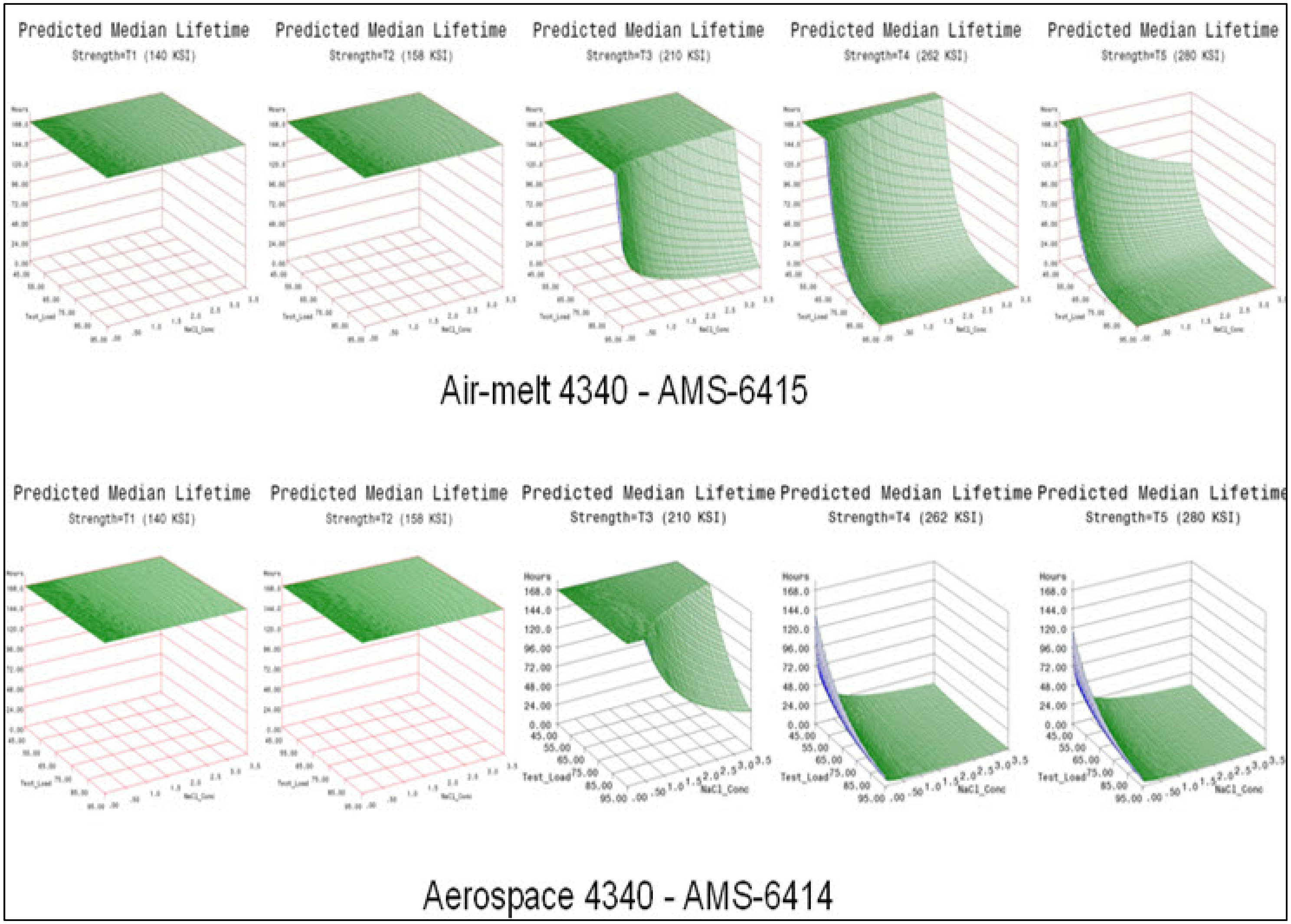

Figure 11. Final 1d specimen geometry life-prediction models.

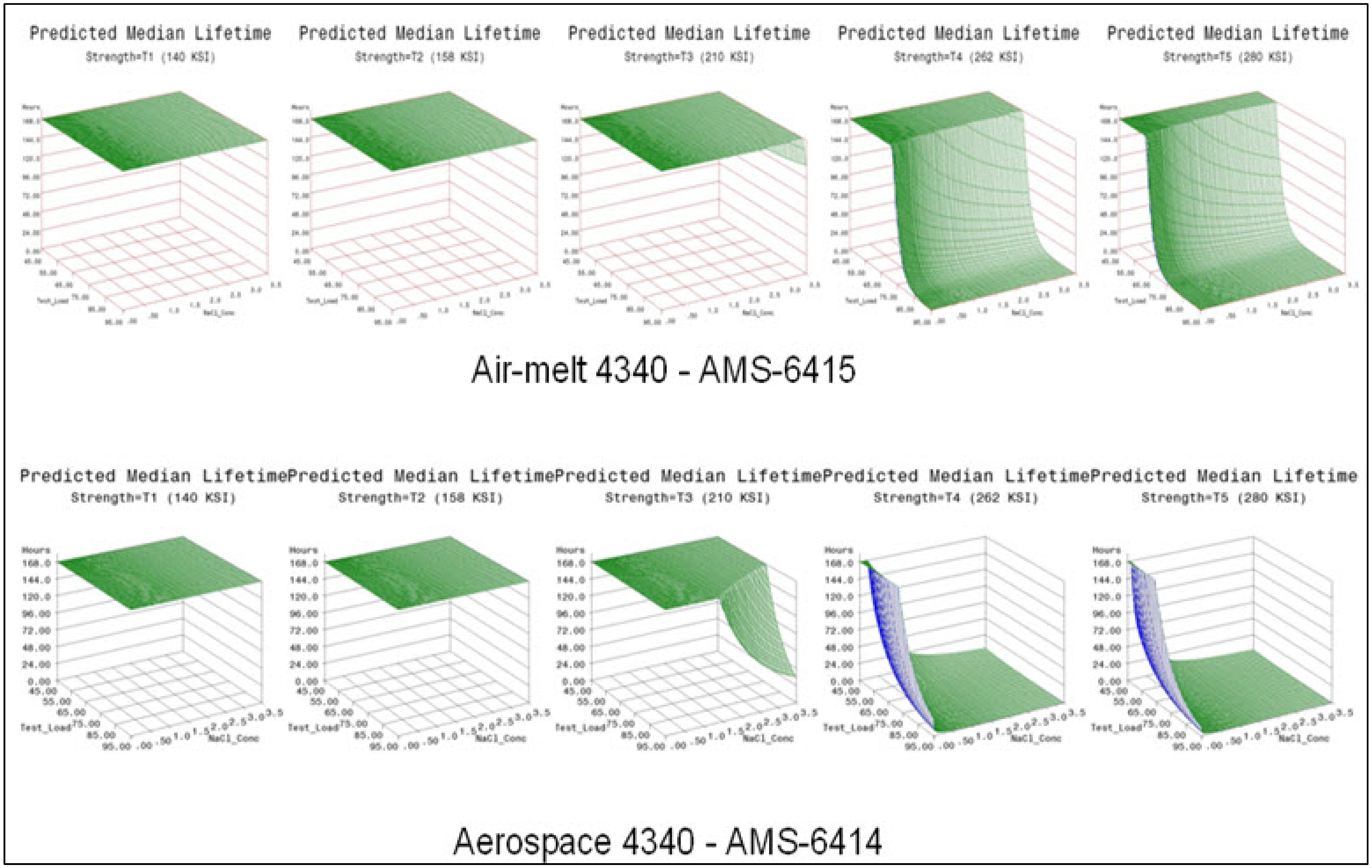

Figure 12. Final 1e specimen geometry life-prediction models.

6. Discussion

It was interesting to discover that air-melted, AMS-6415 steel was more tolerant than aerospace grade, AMS-6414 steel. It has long been held that air-melted steel was the weakest link. Indeed, it has lower tensile strength at equivalent hardness. Intuitively this makes some sense, since hardness is determined in a compressive test, while tensile strength is performed in tension. The weak inclusion points would most likely reveal themselves preferentially in tension. The general thinking in hydrogen sensitivity is that if you test the worst case in a specific environment and your material survives, then all other cases will not fail. With air-melted 4340 steel known to be inferior to aerospace grade 4340 in terms of tensile strength, it was used as the performance gage material.

Across all geometries, it can be observed in the data that air-melted steel demonstrated more tolerance to hydrogen. This was not expected. All other conditions in the testing protocol were identical. The environment and the loading method were the same; the heat treatment was performed at the same time in the same ovens. The only difference was the material grade of the steel. The case may be made that the cleanliness of aerospace-grade steel removes the flaws in the atomic lattice where hydrogen migrates. The less imperfections that the lattice has, the less distributed the damaging hydrogen is. If one assumes that an equivalent amount of hydrogen enters both materials under similar conditions, then one must conclude that the flaws within the air-melted grade lower the sensitivity of that material to hydrogen because each flaw has the ability to hold some hydrogen as a trap. In the aerospace material with limited inclusions, the hydrogen has nowhere to be absorbed and hide, and therefore migrates to the next largest site, the grain boundaries. When the hydrogen concentration in a load-stressed area goes beyond a critical threshold, the grain boundaries tear apart. This is why hydrogen embrittlement failures exhibit blocky brittle fracture surfaces representative of individual grain boundaries.

Since this type of predictive model had never been attempted before to assess hydrogen sensitivity, the results were extremely satisfying. The predictive models express hydrogen sensitivity in terms of applied load, material strength, and hydrogen environment. In this case, the hydrogen environment is a representation of the natural environmental corrosion cycle. In terms of NaCl salt concentration, 3.5% is widely accepted to be the worst-case scenario for corrosion of steel. Values higher than 3.5% actually result in a lower corrosion rate. The time duration, 168 h (1 week), is above what is accepted as the lifetime cutoff for service environments, 150 h. Essentially, this data suggests that if the material demonstrates no hydrogen sensitivity in a 3.5% salt-concentration environment for 168 h at a specific strength and applied-load combination, then it should not be expected to fail in a lifetime of service exposure in our natural environment at that strength and applied load level. The “safe zone” in the graphical representation of the models is the area below the curves.

By comparing all of the models across test geometry, it can be seen that the 3.5% NaCl is not a severe enough environment to cause hydrogen embrittlement at or below the 158 ksi strength level. All of the “T1, 140 ksi” and “T2, 158 ksi” strength levels are flat, showing no sensitivity. This does not mean that in an environment that emits more hydrogen, no sensitivity would be expected. The converse is actually true, industrial processes like electroplating, or acidic or alkaline cleaning would certainly be expected to show sensitivity to hydrogen at or below the 158-ksi material strength level.

Although varying performance can be observed across test geometry, the trends are certainly in agreement. The sensitivity increases with material strength level, applied load, and to a lesser degree, with NaCl concentration. All of these trends are in line with traditional expectations. While material strength level is typically given consideration with regard to hydrogen sensitivity, applied load is often forgotten. Residual stresses from forming, quenching, or from assembly can often reach 40%–45% of the ultimate tensile strength (UTS). This is important to remember because these life-prediction models show sensitivity beginning at, or even below, that region. This supports traditional findings where components sometimes break on the shelf while waiting to be placed in service. When combined with a design stress or inservice applied stress, catastrophic failure is much more likely to occur. The degree of heightened sensitivity from applied stress was unknown until now because it has never been investigated.

It can also be observed in the data that the 1d geometry shows the highest sensitivity. It has the highest stress intensity, stemming from the smallest notch-root radius. It also has historically performed in comparative tests with heightened sensitivity. While this test geometry may not be representative of every application in terms of stress intensity, one would be able to apply a factor of safety to this life-prediction model and have confidence that a similar application would not fail due to hydrogen embrittlement. All of the models have similar trends, but a risk analysis would likely be developed from a worst case and not middle of the pack performance. The 1d geometry is also a self-loading geometry, so it is conducive to testing in various environments since no mechanical test frame is needed. This specimen geometry will be used in subsequent maintenance fluid and coatings work evaluating hydrogen sensitivity.

7. Conclusions

The following conclusions can be drawn from the data developed from this work.

1. Life-prediction models were developed that accurately represent the expected hydrogen sensitivity over the range of parameters explored for air-melted and aerospace grade 4340 steel.

2. Air-melted 4340 steel has less sensitivity to hydrogen than aerospace grade 4340 steel.

3. The trends observed in the data were reasonably consistent across all test geometries. Sensitivity increases with applied load, material strength, and to a lesser degree NaCl concentration.

4. Applied stress has the most direct effect on hydrogen sensitivity, while material strength is a close second. Increasing the value of either parameter directly heightens the sensitivity to hydrogen.

5. Air-melted and aerospace 4340 steel does not appear susceptible to hydrogen absorbed from environmental corrosion below the 160-ksi strength level.

6. High-residual stress levels (40%–50% of UTS) are capable of causing hydrogen embrittlement without further applied-system stresses.

7. The 1d test geometry proved the most sensitive to hydrogen and also conducive to testing multiple specimens in various environments without requiring test load frames.

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List of Symbols, Abbreviations, and Acronyms

ARL	U.S. Army Research Laboratory
ASTM	American Society for Testing and Materials
DoE	Design of Experiment
h	hour (s)
HE	hydrogen embrittlement
LHE	low hydrogen embrittlement
NaCl	sodium chloride
NFS	notch fracture strength
SERDP	Strategic Environment Research and Development Program
UTS	ultimate tensile strength
wt.%	weight-percent

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